

1-(3,5-Difluorophenyl)-4,4,4-trifluorobutane-1,3-dione

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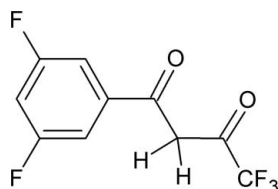
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Key indicators: single-crystal X-ray study; $T = 294$ K; mean $\sigma(\text{C}-\text{C}) = 0.006$ Å; R factor = 0.074; wR factor = 0.239; data-to-parameter ratio = 10.4.

In the title compound, $\text{C}_{10}\text{H}_5\text{F}_5\text{O}_2$, the $\text{C}=\text{O}$ bonds are *syn* to one another. In the crystal, molecules are linked into $C(9)$ chains parallel to $[101]$ through weak $\text{C}-\text{H}\cdots\text{O}$ interactions, with the O atom adjacent to the $-\text{CF}_3$ group acting as the acceptor.

Related literature

For biological-activity studies of compounds with trifluoromethyl substituents, see: Manoj Kumar *et al.* (2013).



Experimental

Crystal data

$\text{C}_{10}\text{H}_5\text{F}_5\text{O}_2$

$M_r = 252.14$

Monoclinic, $C2/c$
 $a = 12.393$ (4) Å
 $b = 13.433$ (5) Å
 $c = 12.877$ (5) Å
 $\beta = 112.49$ (2)°
 $V = 1980.7$ (13) Å³

$Z = 8$
Mo $K\alpha$ radiation
 $\mu = 0.18$ mm⁻¹
 $T = 294$ K
 $0.24 \times 0.20 \times 0.16$ mm

Data collection

Bruker APEXII CCD
diffractometer
Absorption correction: multi-scan
(*SADABS*; Bruker, 2009)
 $T_{\min} = 0.959$, $T_{\max} = 0.972$

5651 measured reflections
1604 independent reflections
1246 reflections with $I > 2\sigma(I)$
 $R_{\text{int}} = 0.040$

Refinement

$R[F^2 > 2\sigma(F^2)] = 0.074$
 $wR(F^2) = 0.239$
 $S = 1.08$
1604 reflections

154 parameters
H-atom parameters constrained
 $\Delta\rho_{\text{max}} = 0.43$ e Å⁻³
 $\Delta\rho_{\text{min}} = -0.47$ e Å⁻³

Table 1

Hydrogen-bond geometry (Å, °).

| $D-H\cdots A$ | $D-H$ | $H\cdots A$ | $D\cdots A$ | $D-H\cdots A$ |
|--|-------|-------------|-------------|---------------|
| $\text{C3}-\text{H3}\cdots\text{O2}^i$ | 0.93 | 2.53 | 3.462 (5) | 177 |

Symmetry code: (i) $x - \frac{1}{2}, -y + \frac{1}{2}, z + \frac{1}{2}$.

Data collection: *APEX2* (Bruker, 2009); cell refinement: *SAINT-Plus* (Bruker, 2009); data reduction: *SAINT-Plus*; program(s) used to solve structure: *SHELXS97* (Sheldrick, 2008); program(s) used to refine structure: *SHELXL97* (Sheldrick, 2008); molecular graphics: *Mercury* (Macrae *et al.*, 2008); software used to prepare material for publication: *SHELXL97*.

The authors acknowledge the IOE X-ray diffractometer Facility, University of Mysore, Mysore, for the data collection. KEM and SS acknowledge Tumkur University, Tumkur, for providing laboratory facilities.

Supplementary data and figures for this paper are available from the IUCr electronic archives (Reference: HB7151).

References

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supporting information

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S1. Comment

As part of our ongoing studies of the biological activities of compounds with a trifluoromethyl substituent (Manoj Kumar *et al.*, 2013), we now describe the structure of the title compound, (I).

The two C=O bonds are *syn* to one another (Fig.1), the O1—C7—C8—C9 and O2—C9—C8—C7 torsion angles being 0.4 (5) and -0.3 (6)°, respectively. In the crystal, the molecules are linked into C(9) chains parallel to [101] through a weak C3—H3···O2 interaction (Fig.2).

S2. Experimental

3,5-Difluoroacetophenone (1 mmol) and sodium hydride (1.5 mmol) were taken in dry THF (20 ml), and the solution was stirred for 30 min at 0°C. To this solution trifluoroethylacetate (1.2 mmol) was added and the reaction mixture was stirred for 10 h at room temperature under nitrogen atmosphere. The reaction was monitored by TLC. The crude mass was purified by column chromatography using petroleum ether and ethyl acetate as an eluent (7:3), to obtain a yellow solid. Pale yellow prisms were obtained by recrystallisation from dichloromethane/methanol (9:1) solution.

S3. Refinement

The H atoms were positioned with idealized geometry using a riding model with C—H = 0.93–0.97 Å. All H atoms were refined with isotropic displacement parameters (set to 1.2 times of the U_{eq} of the parent atom).

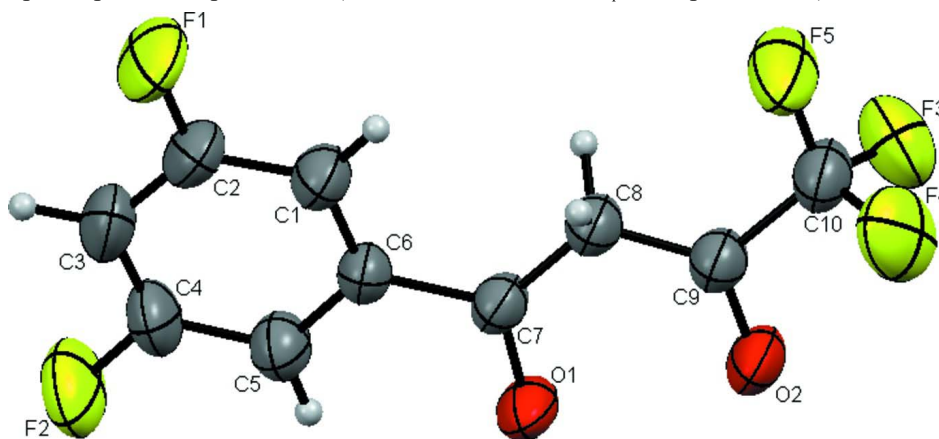
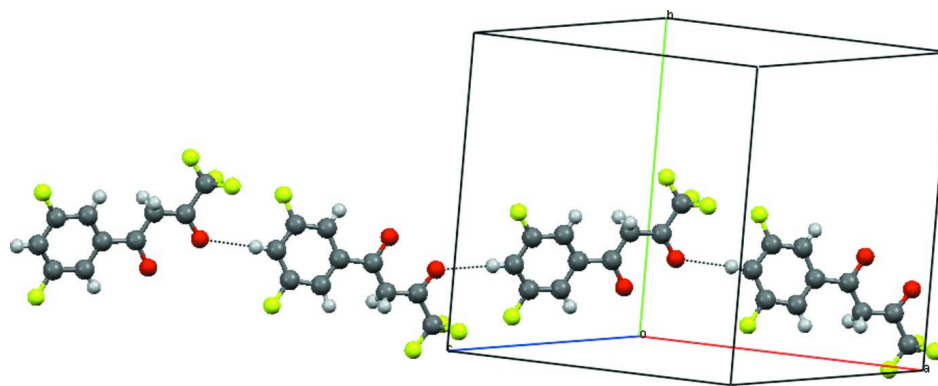


Figure 1

Molecular structure of the title compound, showing displacement ellipsoids drawn at the 50% probability level.

**Figure 2**

Molecular packing forming C(9) chains parallel to [101] with hydrogen bonding shown as dashed lines.

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Crystal data

$C_{10}H_5F_5O_2$
 $M_r = 252.14$
 Monoclinic, $C2/c$
 Hall symbol: $-C\ 2yc$
 $a = 12.393\ (4)\ \text{\AA}$
 $b = 13.433\ (5)\ \text{\AA}$
 $c = 12.877\ (5)\ \text{\AA}$
 $\beta = 112.49\ (2)^\circ$
 $V = 1980.7\ (13)\ \text{\AA}^3$
 $Z = 8$
 $F(000) = 1008$

Prism
 $D_x = 1.691\ \text{Mg m}^{-3}$
 Melting point: 393 K
 Mo $K\alpha$ radiation, $\lambda = 0.71073\ \text{\AA}$
 Cell parameters from 1023 reflections
 $\theta = 0.0\text{--}24.6^\circ$
 $\mu = 0.18\ \text{mm}^{-1}$
 $T = 294\ \text{K}$
 Prism, colourless
 $0.24 \times 0.20 \times 0.16\ \text{mm}$

Data collection

Bruker APEXII CCD
 diffractometer
 Radiation source: fine-focus sealed tube
 Graphite monochromator
 ϕ and ω scans
 Absorption correction: multi-scan
 (*SADABS*; Bruker, 2009)
 $T_{\min} = 0.959$, $T_{\max} = 0.972$

5651 measured reflections
 1604 independent reflections
 1246 reflections with $I > 2\sigma(I)$
 $R_{\text{int}} = 0.040$
 $\theta_{\max} = 24.6^\circ$, $\theta_{\min} = 2.3^\circ$
 $h = -14 \rightarrow 14$
 $k = -15 \rightarrow 7$
 $l = -14 \rightarrow 14$

Refinement

Refinement on F^2
 Least-squares matrix: full
 $R[F^2 > 2\sigma(F^2)] = 0.074$
 $wR(F^2) = 0.239$
 $S = 1.08$
 1604 reflections
 154 parameters
 0 restraints
 Primary atom site location: structure-invariant
 direct methods

Secondary atom site location: difference Fourier
 map
 Hydrogen site location: inferred from
 neighbouring sites
 H-atom parameters constrained
 $w = 1/[\sigma^2(F_o^2) + (0.1433P)^2 + 2.2069P]$
 where $P = (F_o^2 + 2F_c^2)/3$
 $(\Delta/\sigma)_{\max} = 0.012$
 $\Delta\rho_{\max} = 0.43\ \text{e \AA}^{-3}$
 $\Delta\rho_{\min} = -0.47\ \text{e \AA}^{-3}$

Special details

Geometry. All e.s.d.'s (except the e.s.d. in the dihedral angle between two l.s. planes) are estimated using the full covariance matrix. The cell e.s.d.'s are taken into account individually in the estimation of e.s.d.'s in distances, angles and torsion angles; correlations between e.s.d.'s in cell parameters are only used when they are defined by crystal symmetry. An approximate (isotropic) treatment of cell e.s.d.'s is used for estimating e.s.d.'s involving l.s. planes.

Refinement. Refinement of F^2 against ALL reflections. The weighted R -factor wR and goodness of fit S are based on F^2 , conventional R -factors R are based on F , with F set to zero for negative F^2 . The threshold expression of $F^2 > \sigma(F^2)$ is used only for calculating R -factors(gt) etc. and is not relevant to the choice of reflections for refinement. R -factors based on F^2 are statistically about twice as large as those based on F , and R -factors based on ALL data will be even larger.

Fractional atomic coordinates and isotropic or equivalent isotropic displacement parameters (\AA^2)

| | <i>x</i> | <i>y</i> | <i>z</i> | $U_{\text{iso}}^*/U_{\text{eq}}$ |
|-----|-------------|--------------|------------|----------------------------------|
| C1 | 0.0373 (3) | 0.3441 (3) | 0.4970 (3) | 0.0558 (9) |
| H1 | 0.0691 | 0.4058 | 0.4923 | 0.067* |
| C2 | −0.0308 (3) | 0.3323 (3) | 0.5594 (3) | 0.0624 (10) |
| C3 | −0.0787 (4) | 0.2426 (3) | 0.5705 (3) | 0.0684 (11) |
| H3 | −0.1238 | 0.2360 | 0.6135 | 0.082* |
| C4 | −0.0565 (4) | 0.1636 (3) | 0.5147 (4) | 0.0649 (10) |
| C5 | 0.0106 (3) | 0.1700 (3) | 0.4506 (3) | 0.0574 (9) |
| H5 | 0.0242 | 0.1143 | 0.4144 | 0.069* |
| C6 | 0.0574 (3) | 0.2624 (2) | 0.4416 (3) | 0.0474 (8) |
| C7 | 0.1298 (3) | 0.2699 (2) | 0.3734 (3) | 0.0476 (8) |
| C8 | 0.1640 (3) | 0.3603 (3) | 0.3419 (3) | 0.0511 (8) |
| H8A | 0.0937 | 0.3983 | 0.3019 | 0.061* |
| H8B | 0.2077 | 0.3967 | 0.4101 | 0.061* |
| C9 | 0.2304 (3) | 0.3596 (3) | 0.2760 (3) | 0.0515 (9) |
| C10 | 0.2656 (3) | 0.4562 (3) | 0.2375 (3) | 0.0624 (10) |
| F1 | −0.0500 (3) | 0.4121 (2) | 0.6129 (3) | 0.1000 (11) |
| F2 | −0.1027 (3) | 0.0740 (2) | 0.5224 (3) | 0.1010 (11) |
| F3 | 0.2245 (3) | 0.4627 (2) | 0.1272 (2) | 0.0913 (9) |
| F4 | 0.3807 (2) | 0.4629 (2) | 0.2695 (3) | 0.0989 (10) |
| F5 | 0.2306 (3) | 0.5370 (2) | 0.2748 (3) | 0.1110 (13) |
| O1 | 0.1598 (2) | 0.18650 (19) | 0.3425 (2) | 0.0671 (8) |
| O2 | 0.2645 (2) | 0.2827 (2) | 0.2411 (2) | 0.0666 (8) |

Atomic displacement parameters (\AA^2)

| | U^{11} | U^{22} | U^{33} | U^{12} | U^{13} | U^{23} |
|-----|-------------|-------------|-------------|--------------|-------------|--------------|
| C1 | 0.065 (2) | 0.051 (2) | 0.066 (2) | 0.0018 (15) | 0.0410 (17) | −0.0022 (16) |
| C2 | 0.072 (2) | 0.063 (2) | 0.070 (2) | 0.0110 (18) | 0.0473 (19) | −0.0002 (17) |
| C3 | 0.070 (2) | 0.083 (3) | 0.073 (2) | 0.007 (2) | 0.050 (2) | 0.012 (2) |
| C4 | 0.072 (2) | 0.059 (2) | 0.081 (2) | −0.0095 (18) | 0.048 (2) | 0.0108 (19) |
| C5 | 0.064 (2) | 0.050 (2) | 0.069 (2) | −0.0058 (15) | 0.0380 (17) | −0.0011 (15) |
| C6 | 0.0508 (18) | 0.0460 (18) | 0.0561 (18) | 0.0013 (13) | 0.0323 (15) | 0.0034 (14) |
| C7 | 0.0473 (16) | 0.0457 (18) | 0.0588 (19) | 0.0002 (13) | 0.0306 (15) | −0.0036 (14) |
| C8 | 0.0564 (18) | 0.0448 (19) | 0.065 (2) | 0.0001 (14) | 0.0373 (16) | −0.0009 (15) |
| C9 | 0.0535 (18) | 0.048 (2) | 0.063 (2) | −0.0011 (14) | 0.0335 (16) | −0.0016 (15) |
| C10 | 0.069 (2) | 0.058 (2) | 0.079 (3) | −0.0047 (17) | 0.049 (2) | −0.0035 (18) |

| | | | | | | |
|----|-------------|-------------|-------------|--------------|-------------|--------------|
| F1 | 0.146 (3) | 0.0803 (18) | 0.121 (2) | 0.0129 (16) | 0.105 (2) | −0.0090 (15) |
| F2 | 0.125 (2) | 0.0773 (18) | 0.138 (3) | −0.0305 (16) | 0.092 (2) | 0.0011 (16) |
| F3 | 0.112 (2) | 0.0859 (19) | 0.0818 (18) | −0.0116 (15) | 0.0440 (15) | 0.0191 (13) |
| F4 | 0.0676 (15) | 0.096 (2) | 0.139 (3) | −0.0226 (13) | 0.0460 (15) | 0.0177 (17) |
| F5 | 0.169 (3) | 0.0515 (16) | 0.173 (3) | −0.0089 (16) | 0.133 (3) | −0.0034 (16) |
| O1 | 0.0897 (19) | 0.0399 (14) | 0.102 (2) | 0.0042 (12) | 0.0704 (16) | −0.0031 (12) |
| O2 | 0.0793 (17) | 0.0555 (16) | 0.0924 (19) | 0.0030 (12) | 0.0633 (15) | −0.0029 (13) |

Geometric parameters (Å, °)

| | | | |
|-------------|------------|--------------|------------|
| C1—C2 | 1.379 (5) | C6—C7 | 1.479 (5) |
| C1—C6 | 1.383 (5) | C7—O1 | 1.290 (4) |
| C1—H1 | 0.9300 | C7—C8 | 1.396 (5) |
| C2—F1 | 1.344 (5) | C8—C9 | 1.389 (5) |
| C2—C3 | 1.374 (6) | C8—H8A | 0.9700 |
| C3—C4 | 1.367 (6) | C8—H8B | 0.9700 |
| C3—H3 | 0.9300 | C9—O2 | 1.262 (4) |
| C4—F2 | 1.353 (5) | C9—C10 | 1.512 (5) |
| C4—C5 | 1.381 (5) | C10—F3 | 1.316 (5) |
| C5—C6 | 1.393 (5) | C10—F5 | 1.327 (5) |
| C5—H5 | 0.9300 | C10—F4 | 1.328 (4) |
| C2—C1—C6 | 118.8 (3) | O1—C7—C8 | 120.7 (3) |
| C2—C1—H1 | 120.6 | O1—C7—C6 | 115.8 (3) |
| C6—C1—H1 | 120.6 | C8—C7—C6 | 123.5 (3) |
| F1—C2—C3 | 118.5 (3) | C9—C8—C7 | 119.2 (3) |
| F1—C2—C1 | 118.4 (4) | C9—C8—H8A | 107.5 |
| C3—C2—C1 | 123.1 (4) | C7—C8—H8A | 107.5 |
| C4—C3—C2 | 116.4 (3) | C9—C8—H8B | 107.5 |
| C4—C3—H3 | 121.8 | C7—C8—H8B | 107.5 |
| C2—C3—H3 | 121.8 | H8A—C8—H8B | 107.0 |
| F2—C4—C3 | 118.5 (3) | O2—C9—C8 | 125.5 (3) |
| F2—C4—C5 | 117.9 (4) | O2—C9—C10 | 114.0 (3) |
| C3—C4—C5 | 123.7 (4) | C8—C9—C10 | 120.5 (3) |
| C4—C5—C6 | 118.0 (4) | F3—C10—F5 | 106.9 (4) |
| C4—C5—H5 | 121.0 | F3—C10—F4 | 104.9 (3) |
| C6—C5—H5 | 121.0 | F5—C10—F4 | 107.1 (3) |
| C1—C6—C5 | 120.0 (3) | F3—C10—C9 | 111.8 (3) |
| C1—C6—C7 | 121.5 (3) | F5—C10—C9 | 114.1 (3) |
| C5—C6—C7 | 118.4 (3) | F4—C10—C9 | 111.5 (3) |
| C6—C1—C2—F1 | −179.9 (3) | C5—C6—C7—O1 | −11.0 (5) |
| C6—C1—C2—C3 | 0.8 (6) | C1—C6—C7—C8 | −12.7 (5) |
| F1—C2—C3—C4 | −179.9 (4) | C5—C6—C7—C8 | 168.3 (3) |
| C1—C2—C3—C4 | −0.6 (6) | O1—C7—C8—C9 | 0.4 (5) |
| C2—C3—C4—F2 | −179.5 (4) | C6—C7—C8—C9 | −178.9 (3) |
| C2—C3—C4—C5 | 0.4 (6) | C7—C8—C9—O2 | −0.3 (6) |
| F2—C4—C5—C6 | 179.4 (3) | C7—C8—C9—C10 | 178.1 (3) |

| | | | |
|-------------|------------|--------------|------------|
| C3—C4—C5—C6 | −0.5 (6) | O2—C9—C10—F3 | 59.0 (4) |
| C2—C1—C6—C5 | −0.9 (5) | C8—C9—C10—F3 | −119.5 (4) |
| C2—C1—C6—C7 | −179.9 (3) | O2—C9—C10—F5 | −179.5 (3) |
| C4—C5—C6—C1 | 0.7 (5) | C8—C9—C10—F5 | 1.9 (5) |
| C4—C5—C6—C7 | 179.8 (3) | O2—C9—C10—F4 | −58.1 (4) |
| C1—C6—C7—O1 | 168.0 (3) | C8—C9—C10—F4 | 123.4 (4) |

Hydrogen-bond geometry (Å, °)

| <i>D</i> —H \cdots <i>A</i> | <i>D</i> —H | H \cdots <i>A</i> | <i>D</i> \cdots <i>A</i> | <i>D</i> —H \cdots <i>A</i> |
|--------------------------------|-------------|---------------------|----------------------------|-------------------------------|
| C3—H3 \cdots O2 ⁱ | 0.93 | 2.53 | 3.462 (5) | 177 |

Symmetry code: (i) $x-1/2, -y+1/2, z+1/2$.